INTERNATIONAL STANDARD

ISO

CD 14624-6

Committee Draft

2001-04-01

Space systems ¾ Safety and compatibility of materials – Part 6: Test method for determining the reactivity of processing materials with aerospace fluids

Systémes spatiaux – Sécurite et compatibilité des materiaux – Partie 6: Methode d'essai pour determination de la reactivité des materiaux avec fluides spatiaux

Reference number

ISO CD 14624-6:2001(E)

Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and nongovernmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14624-6 was prepared by Technical Committee ISO/TC 20, Aircraft and Space Vehicles; Sub-Committee SC 14, Space Systems and Operations.

ISO CD 14624 consists of the following parts, under the general title Space systems – Safety and compatibility of materials:

Part 1: Test method for upward flammability of materials

Part 2: Test method for determination of electrical wire insulation and accessory flammability

Part 3: Test method for determination of offgassed products from materials and assembled articles

Part 4: Test method for upward flammability of materials in gaseous oxygen and oxygen-enriched environments

Part 5: Test method for determining the reactivity of system/component materials with aerospace hypergolic propellants

Part 6: Test method for determining the reactivity of processing materials with aerospace fluids

Part 7: Test method for determining the permeability and penetration of materials to aerospace fluids

Introduction

This purpose of this International Standard is to identify changes resulting from exposure of a material to an aerospace fluid that renders either the material unsuitable for use or produces an exothermic reaction that may result in a fire.

Space systems ¾ Safety and compatibility of materials – Part 6: Test method for determining the reactivity of processing materials with aerospace fluids

1 Scope

This International Standard describes test equipment and techniques used to identify interactions resulting from exposure of a material to an aerospace fluid.

This International Standard may be used to determine the reactivity of processing materials with aerospace fluids either through intent or casual exposure. This International Standard provides a means to determine the effects of minor amounts of fluids, such as a splash or spill, on materials used in ground support processing operations.

2 Normative references

The following normative references contain provisions that, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subjects to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4599:1986, Plastics – Determination of resistance to environmental stress cracking (ESC) – Bent strip method.

ISO 4954:1993, Steels for cold heading and cold extruding.

ISO 14951-3:1999, Space systems — Fluid characteristics — Part 3: Nitrogen.

3 Definitions

For the purposes of this International Standard, the following terms and definitions shall apply:

3.1
aerospace fluids
fluids that are commonly used in the fabrication, development, and processing of materials and in the production of aerospace and ground support equipment and propellants

EXAMPLE Cleaning agents, lubricants, and solvents are examples of aerospace processing fluids.

3.2
aerospace materials
materials used in the fabrication and/or production of ground support and flight components and systems

3.3
degradation
an adverse physical or chemical change in a substance

3.4
exothermic reaction
a chemical reaction that generates heat

3.5
ground support equipment
equipment used in the processing and preparation of flight hardware

3.6

immersion test
a test in which the fluid covers the entire sample for the duration of the test

3.7
reaction
a chemical change in which a substance decomposes, combines with other substances, or interchanges constituents with other substances

3.8
test conclusions
those results that are reported on the reactivity test report form

4 Safety precautions

4.1 Laboratory facilities

Many aerospace fluids are considered to be toxic chemicals. These chemicals shall only be opened and used inside an approved laboratory hood.

4.2 Protective clothing

Personal protective clothing shall be worn by personnel when performing these tests. The minimum protections required are fluid-compatible gloves, laboratory apron, and face shield or goggles.

5 Test procedure

5.1 Sample receiving inspection

When received, the test material must be accompanied by proper identification. The minimum information required is the manufacturer, trade name, composition, specification, generic name, and batch/lot number. A visual inspection shall be performed and any anomalies shall be noted. Figure 2 is a suitable material identification form.

5.2 Sample preparation

5.2.1 General

The sample should be tested in the intended use form (such as sheets or foams) and in the as-received thickness. Samples for the immersion test should have a surface area of 250 $\pm$ 5 square millimetres (mm^2).

5.2.2 Sample cleaning

Samples shall be cleaned and dried to the end-use specifications. Contamination on the surfaces of solid, nonporous samples shall be removed by washing with deionized water and mild detergent, rinsing with deionized water, and drying with filtered, gaseous nitrogen. Particulate on the surfaces of solid, porous samples shall be removed with filtered, gaseous nitrogen meeting the requirements of ISO 14951-3.

5.2.3 Sample inspection

The sample shall be inspected to ensure it is at the specified worst-case thickness. Flaws and any residual contamination shall be noted. If the flaws result from sample preparation at the test facility, new samples shall be prepared. Samples with flaws that inordinately increase the surface area to bulk mass ratios should not be tested. Samples shall be weighed and individually identified.

5.2.4 Test sample configuration

5.2.4.1 Sheets, film, and fabrics

Material being tested for chemical reactivity should be cut in the form of a 100-mm square sample in the use thickness (see Figure 3). To determine changes in the mechanical properties of a material, the sample shall be cut in the form of a 100 mm by 150 mm rectangle (see Figure 3). For nonisotropic materials, the 100 mm by 150 mm samples shall be cut from both the machine (warp) and transverse (fill) directions.

REACTIVITY TEST REPORT FORM

		Date	Sample Number
Requesting Organization	Requestor	Telephone No.	Reference Document
Vehicle	System		

Material Name or Manufacturer's ID

Chemical Class of Material

Generic Name of Material

Special Instructions

TEST CONDITIONS

Test 1 Sample per: _____

Test Name: _____

Test Fluid: _____

TEST DATA

Test Sample Description: _____

Material Dimensions: _____

Media Volume (ml): _____

Media Exposure Time (min): _____

OBSERVATIONS

Burn _____ Temp. Change _____

Smoke _____ Soluble _____

Froth _____ Fracture _____

Bubble _____ Swell _____

Char _____

Remarks: _____

VISUAL CHARACTERISTICS

	Pretest	Posttest
Color		
Opaque		
Translucent		
Transparent		

Remarks: _____

BULK CHARACTERISTICS

	Pretest	Posttest
Shape		
Flexible		
Rigid		
Soft		
Hard		
Friable		
Powder		

Remarks: _____

SURFACE CHARACTERISTICS

	Pretest	Posttest
Smooth		
Rough		
Wrinkled		
Pitted		
Woven		
Matted		
Tacky		

Remarks: _____

Other Observations

Conclusions

☐ No Significant Reactivity Observed

☐ Slight to Moderate Reactivity Observed

☐ Sample Shows Indications of Gross Incompatibility

Analyst:	Date:	Approval:

Figure 1 - Reactivity test report form

Table 1 – Material identification form

Test Material

Manufacturer _____

Trade Name _____

Composition _____

Specification _____

Generic Name _____

Batch / Lot Number _____

Use Temperature (minimum) _____

Use Temperature (maximum) _____

Fluid Exposure Time (field use) _____

Manufacturer

Name

Address _____

City _____

State _____

Country _____

Supplier

Name

Address _____

City _____

State _____

Country _____

Remarks _____

_____

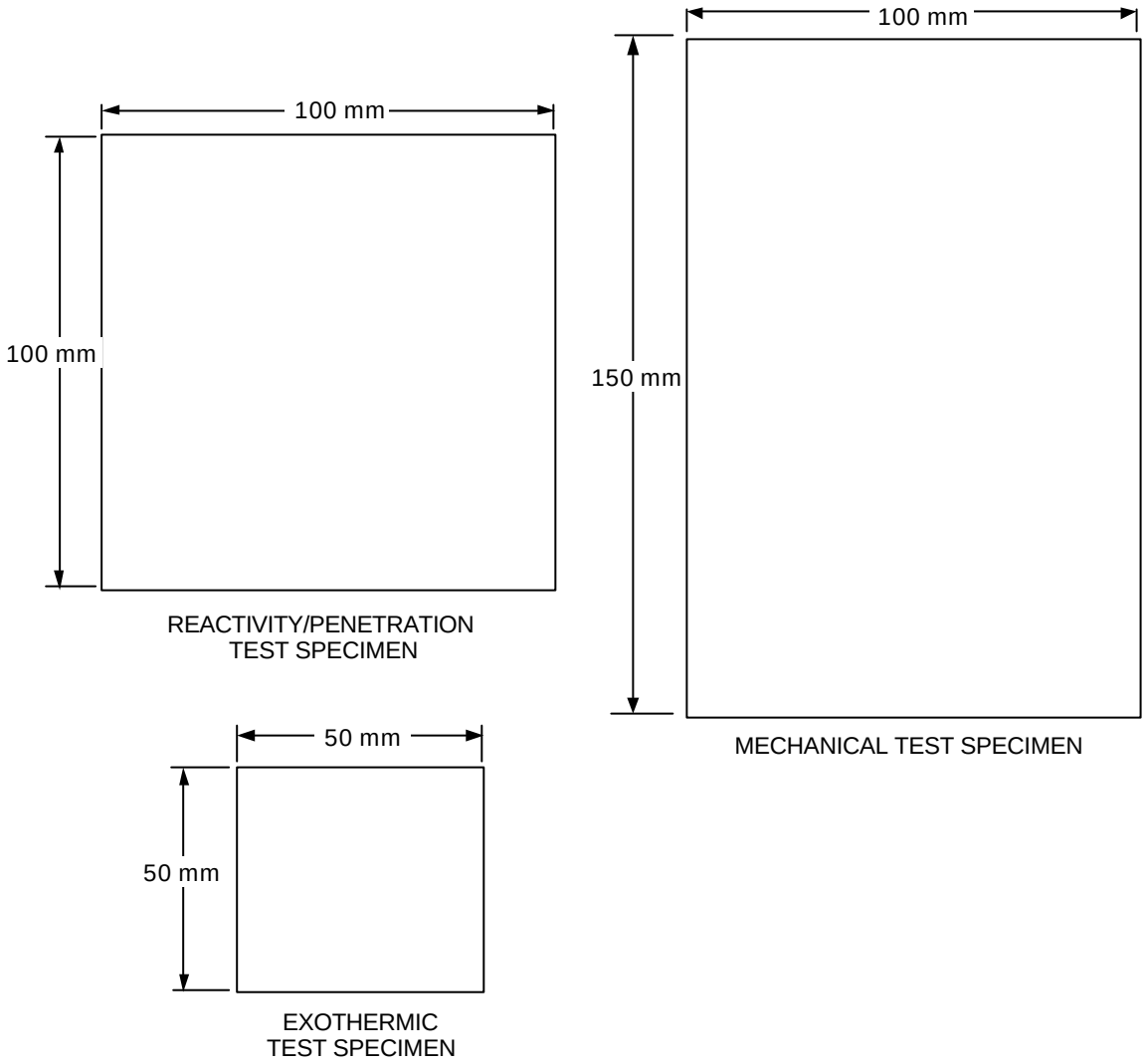

Figure 2 – Test specimen dimensions (millimetres)

5.2.4.2 Adhesives and coatings

Adhesives and coatings shall be applied in a thickness equivalent to normal use on aluminum foil and cured, if necessary, in accordance with the manufacturer's instructions.

5.2.4.3 Tapes

Tapes shall be applied on aluminum foil, a watch glass, or petri dish in the as-received condition and thickness.

5.2.4.4 Greases and gels

Greases and gels shall be applied on aluminum foil, watch glass, or petri dish in a thickness equivalent to normal use and cured, if required, in accordance with the manufacturer's instructions.

5.2.4.5 Liquids

Liquids shall be tested by placing 1 millilitre (mL) in the bottom of a 20 mL glass laboratory beaker.

5.2.4.6 Nonstandard configurations

Complex shapes such as O-rings, cables, pipes, etc., shall be tested in a configuration consistent with the intended use. Samples shall be cleaned as specified in subclause 5.2.2.

5.3 Test methods

5.3.1 Reactivity test

5.3.1.1 General

This test is used to determine a possible material reaction and/or degradation when exposed to propellants or other liquids of interest.

5.3.1.2 Test procedure

The test procedure shall be as follows:

a) Place a test specimen sample of the test material (see Figure 3) on a watch glass or in a petri dish and place a thermocouple in contact with the middle of the specimen. The thermocouple should be jacketed with glass or other inert material to minimize any reaction with the test fluid or the material being tested. Since the evaporation of the test fluid may mask a reaction, a differential thermocouple scheme using one thermocouple in contact with the sample and another thermocouple in contact with the test fluid may be more definitive.

b) Add the test fluid, approximately 1 mL of the specified test fluid, to the center of the sample, taking care not to expose the edges of the sample to the fluid to prevent wicking.

c) Allow the test fluid to stand on the sample for the specified exposure time.

d) Add test fluid as required to maintain a liquid film on the test sample during the specified exposure time.

e) Carefully observe the test sample and thermocouple readout throughout the duration of the test.

f) At the end of the specified exposure time, carefully blot the liquid from the sample and rinse the sample with running water for 60 seconds. Blotting and rinsing should be performed with cognizance of the blotting material compatibility of the test fluid. Rinsing should be performed with cognizance of the environmental regulations governing disposal of the test fluid.

g) Allow the test sample to dry under flowing air or nitrogen for 24 hours prior to final evaluation.

5.3.1.3 Report

The report shall consist of the following as a minimum (an example of a suitable form for reporting the results of this test is shown in Figure 1):

a) The name of the test material, supplier, and manufacturer.

b) The test temperature, any temperature change, duration, and sample thickness before and after the test.

c) Any reactivity observed during the exposure such as burning, smoking, bubbling, frothing, charring, solubility, swelling, or fracture of the sample.

d) Any changes in the condition of the sample after the exposure such as color, flexibility, rigidity, surface condition, transparency, pitting, hardness, tackiness, friability, or powder formation.

e) Test conclusions.

5.3.2 Penetration test

5.3.2.1 General

This test is used to determine both the possible liquid penetration and chemical reactivity of materials when exposed to aerospace fluids or other chemicals of interest. This test should not be performed if the material failed the reactivity test.

5.3.2.2 Test procedure

The test procedure shall be as follows:

a) Place an appropriately sized sample of the test material (see Figure 3) over a beaker.

b) Add approximately 1 mL of the specified test fluid to the center of the sample, taking care not to expose the edges of the sample to the fluid and start the stopwatch.

c) Allow the test fluid to stand on the sample for the specified exposure time.

d) Add test fluid as required to maintain a liquid film on the test sample during the specified exposure time.

e) Carefully observe for the first fallen droplet at the bottom of the beaker and note the time of occurrence.

f) For materials used for protective garments, observe for initial wetness underneath the test sample and note the time of occurrence.

NOTE : Atmospheric condensation could occasionally form underneath a sample during a test, giving a false indication of penetration. In such an event, verification can be made by applying a hypergol compatible blotter that is known to discolor when in contact with a hypergolic fluid.

g) Carefully blot the liquid from the sample at the end of the specified exposure time.

h) Rinse the sample with running water for 60 seconds. Rinsing should be performed with cognizance of the environmental regulations governing disposal of the test fluid.

i) Allow the test sample to dry under flowing air or nitrogen for 24 hours prior to final evaluation.

5.3.2.3 Report

The report shall consist of the following as a minimum (an example of a suitable form for reporting the results of this test is shown in Figure 1):

a) The name of the test material, supplier, and manufacturer.

b) The test temperature, pressure, duration, and sample thickness before and after the test.

c) Any penetration observed during the exposure and the elapsed time of occurrence.

d) For materials used for protective garments, any wetness observed underneath the test sample during the exposure and the elapsed time of occurrence.

e) Any reactivity observed during the exposure such as burning, smoking, bubbling, frothing, charring, solubility, swelling, or fracture of the sample.

f) Any changes in the condition of the sample after the exposure such as color, flexibility, rigidity, surface condition, transparency, pitting, hardness, tackiness, friability, or powder formation.

g) Test conclusions.

5.3.3 Mechanical evaluation test

5.3.3.1 General

This test is used to determine possible changes in the mechanical properties of a material due to exposure to aerospace fluids or other chemicals of interest.

NOTE: The reactivity test (subclause 5.3.1) should be used as a screening test prior to the performance of this test.

5.3.3.2 Test procedure

The test procedure shall be as follows:

a) Select the desired mechanical property to be evaluated and determine the value of that property on unexposed samples as a control.

b) Place a 100 mm by 150 mm test specimen on a flat polytetrafluoroethylene or stainless steel base plate meeting the requirements of ISO 4954 (see Figure 3).

c) Take a template, shown in Figure 4, and apply a bead of fluid-compatible (e.g., perfluoroether) grease around the 75 mm by 25 mm opening (to prevent wicking under the template). An alternative is to have a compatible template constructed with a continuous ridge around the opening on the side facing the sample that may be clamped against the sample effecting a chevron seal.

d) Then clamp both the template (with the grease against the test specimen) and test specimen to the base plate. The test specimen shall be sandwiched between the template and the base plate.

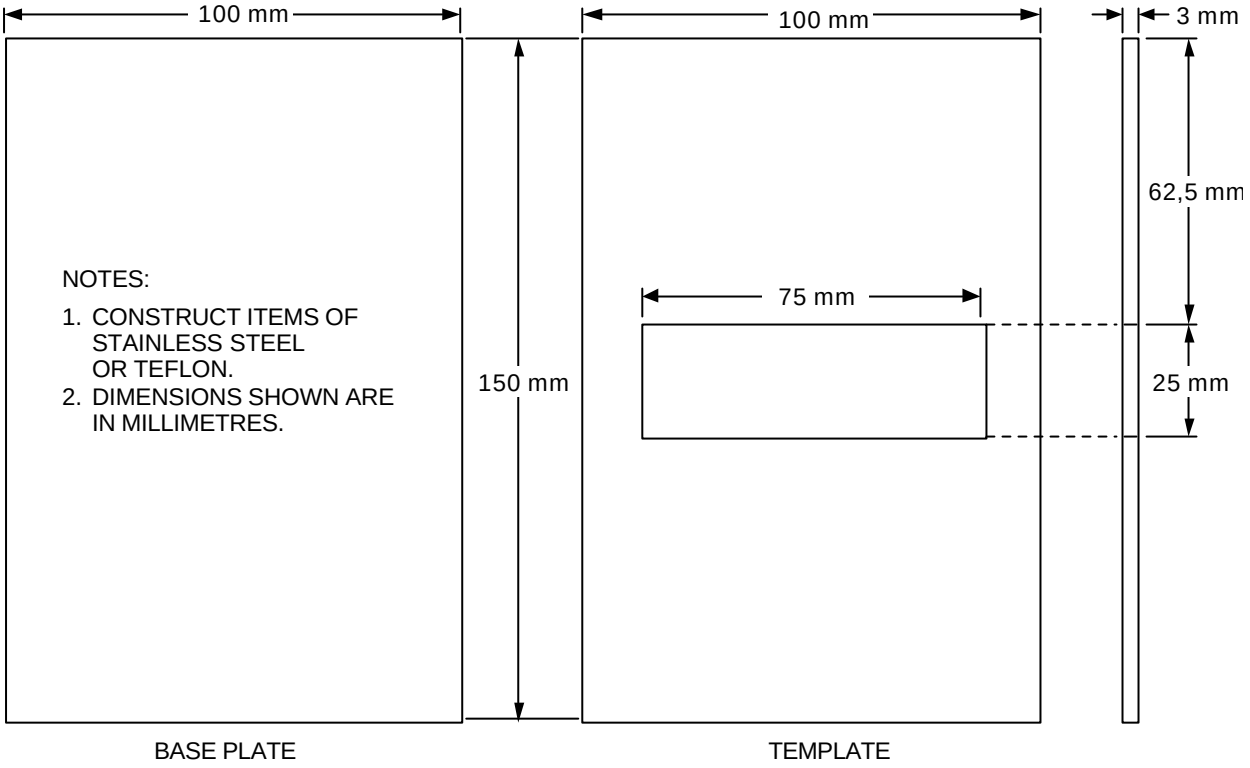

Figure 3 - Exposure fixture for mechanical test

e) Place sufficient test fluid in the slot to wet the full 75 mm by 25 mm exposed strip of test specimen.

f) Allow the test fluid to stand on the test specimen for the specified exposure time.

g) Add test fluid as necessary to maintain a uniform liquid film across the width of the test specimen during the specified exposure time.

h) At the end of the specified exposure time, carefully remove the test fluid and rinse with running water for 60 seconds. Rinsing should be performed with cognizance of the environmental regulations governing disposal of the test fluid.

i) Disassemble the test fixture.

j) If used, wipe the grease off the test specimen.

k) Allow the test sample to dry under flowing air or nitrogen for 24 hours prior to final evaluation.

l) Determine the value of the mechanical property to be evaluated using a test procedure appropriate for the material being evaluated.

5.3.3.3 Report

The report shall consist of the following as a minimum

a) The name of the test material, supplier, and manufacturer.

b) The name of the test chemical.

c) The test temperature, pressure, duration, and sample thickness before and after the test.

d) Any reactivity observed on the test specimen in accordance with subclause 5.3.1.

e) The value of the mechanical property obtained for the exposed and unexposed test material and the method used to measure that property.

5.3.4 Shock sensitivity test

The shock sensitivity of a material exposed to these fluids shall be assessed using mechanical impact test procedures similar to the liquid and gaseous oxygen impact test, drop-weight impact of solid-phase hazardous materials, or the drop-weight impact on monopropellant test.

5.3.5 Exothermic reaction test

5.3.5.1 General

This test shall measure the temperature rise of a material when exposed to aerospace fluids or other chemicals of interest. An excessive temperature rise could suggest an impending, spontaneous ignition of the material.

NOTE: Since nitrogen tetroxide has a boiling point of 20 °C, this method is not applicable for nitrogen tetroxide since its evaporative cooling effect would make any results obtained inconclusive.

5.3.5.2 Test procedure

The test procedure shall be as follows:

a) Take an appropriately sized sample of the test material (see Figure 3) and place it on a watch glass or petri dish.

b) Position a sheathed thermocouple or resistance thermometer such that it touches the center of the sample.

c) Add 1 mL of the specified test fluid to the center of the sample making sure that the sheath is in the liquid.

d) Allow the test fluid to stand on the sample for the specified exposure time while monitoring the temperature.

e) At the end of the specified exposure time, carefully blot the sample dry, rinse with running water for 60 seconds and air dry for 24 hours. Rinsing should be performed with cognizance of the environmental regulations governing disposal of the test fluid.

5.3.5.3 Report

The report shall consist of the following as a minimum (an example of a suitable form for reporting the results of the test is shown in Figure 1).

a) The name of the test material, supplier, and manufacturer.

b) The name of the test chemical.

c) The starting temperature of the test fluid, the maximum fluid temperature observed during the test and the time of occurrence, and the test fluid temperature at the end of the test.

d) The duration of the test and sample dimensions.

e) Any reactivity observed in the test specimen.

5.3.6 Environmental stress cracking test

5.3.6.1 General

ISO 4599 or other appropriate procedures shall be used to determine the susceptibility of a material to environmental stress cracking (ESC) when placed in contact with a test fluid. This procedure utilizes a two-point buckling load technique, which generates a bending stress at the midsection of the test specimen.

Preliminary qualitative tests at relatively high stress levels may be performed to identify materials with a tendency to undergo rapid ESC or crazing and to reduce the total number of tests required to characterize a material. The following tests shall be performed:

a) Perform a series of tests that would determine the time to crack or craze at different stress levels.

b) Plot the stress level versus the time to crack or craze. An L-shaped curve should be obtained.

c) Draw straight lines through the points that make up the vertical and horizontal legs of the L. The intersection of the lines will determine the critical stress level for that particular solvent/material combination.

d) Repeat the procedure for each different solvent/material combination.

5.3.6.2 Report

The report shall contain the following information as a minimum:

a) The name of the test material, supplier, and manufacturer.

b) The name of the test chemical.

c) The test temperature, pressure, duration, and sample thickness (at the exposed area) before and after the test.

d) The length, thickness, and width of the overall test sample.

e) The critical stress level as obtained from the plot.

Bibliography

ASTM D2540-93, Standard Test Method for Drop-Weight Sensitivity of Liquid Monopropellants

ASTM E680-79, Standard Test Method for Drop-Weight Impact Sensitivity of Solid-Phase Hazardous Materials

ASTM G86-98, Standard Test Method for Determining Ignition Sensitivity of Materials to Mechanical Impact in Ambient Liquid Oxygen and Pressurized Liquid and Gaseous Oxygen Environments.

ISO 14951-5:1999, Space systems – Fluid characteristics – Part 5: Nitrogen tetroxide propellant.

ISO 14951-6:1999, Space systems – Fluids characteristics – Part 6: Monomethylhydrazine propellant.

ISO 14951-7:1999, Space systems – Fluid characteristics – Part 7: Hydrazine propellant.

ISO 14951-10:1999, Space systems – Fluid characteristics – Part 10: Water.